DEVELOPMENT OF AN ULTRASONIC VELOCIMETER SYSTEM FOR THE STUDY OF AOUSTIC AND THERMODYNAMIC PROPERTIES OF LIQUIDS

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THESIS

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ABSTRACT

An ultrasonic velocimeter system utilizing two nearly identical velocimeters was developed that could measure the differences in speeds of sound between pure liquids and solutions over the pressure range ambient to 2000 psi and the temperature range ambient to 100°C. Accuracy of two parts in 10^{-6} in frequency corresponding to an average deviation in the speed of sound of .3 cm/sec is demonstrated.

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I. INTRODUCTION

A. PURPOSE

The purpose of this research was to develop an ultrasonic velocimeter system that could be used to obtain precise measurements of the speed of sound and its temperature and pressure derivatives in liquids. Attention is to be focused upon the measurement of differences in properties between pure liquids and solutions. Because of this comparison of properties, two nearly identical velocimeters were developed which could be subjected simultaneously to variations in pressure from ambient to 2000 psi and variations in temperature from ambient to 100°C. Much of the effort of this research was involved in designing, testing, and calibrating the velocimeter system.

Alternately, one velocimeter could have been used to compare the same properties. Such a system would be cycled once through the temperature and pressure range with the pure liquid and unloaded. After reloading with the solution, cycling would be repeated. It would be highly unlikely that the same conditions could be achieved for each datum. Extrapolation and interpolation from the data would be required in making any comparison of properties. Both velocimeters being cycled simultaneously guarantees that essentially the same conditions exist in both delay lines and more specifically that the same point in the equilibrium cycle of each delay line is reached at the same time.

In the design considerations, several guiding and constraining features were evident. The system had to be small enough to be conveniently handled and fitted inside the temperature oven provided for the research. The velocimeters had to be of sufficiently small thermal mass that reasonable time constants could be obtained during temperature cycling. At the same time, the velocimeters had to be designed so that they could be disassembled and reassembled without the need of repeated calibrations.

B. PRINCIPLES OF THE VELOCIMETER

The velocimeter, or acoustic delay line, measures the travel time of a transient wave through a liquid from one end of the velocimeter to the other. The delay line configuration used in this research is similar to those of Greenspan and Tschiegg [1] and Wilson [2] in that it consists of a cylindrical cavity closed on each end by a piezoelectric wafer-like transducer. The speed of sound in a liquid may be accurately determined by filling the cavity with the liquid under investigation and then generating electrical pulses which cause one of the transducers to ring. Each acoustical pulse thus generated reflects back and forth in the liquid between the parallel faces of the transmitting and receiving transducers. The receiving transducer responds to each of the pulses reflecting from it and generates an electrical output which may be displayed on an oscilloscope. Adjustment of the repetition frequency of the pulse generator that excites the transmitting transducer so that the time between successive echoes at the receiver is the same as the time for a round trip between transducers allows superposition of the echoes. The repetition frequency is thus a measure of the speed of sound in the liquid. This relationship is given by $c = 2\ell\nu$ where c is

the speed of sound in the medium and ℓ is the perpendicular distance between the inner faces of the two transducers. Since only the first half cycle of each acoustical pulse corresponds to sound reflected just from the inner faces of the transducers (succeeding half cycles correspond to sound reflected one or more times from an outer face), coincidence is set by maximizing the peak of the first half cycle of the electrical signal representing the superpositioned acoustical echoes. If the first half cycle has too small an amplitude, the second half cycle can be used without affecting the accuracy appreciably: if the same superposition criteria is used for both velocimeters, the errors tend to mutually cancel. This transmission method with its separate transducers for transmitting and receiving has an advantage in that the output of the velocimeter is examined at times different from those at which there is an excitation of the transmitter so that there is no mixing of electrical and acoustical signals [3].

II. EQUIPMENT

(A list of equipment is given on page .)

A. CRYSTAL TRANSDUCERS

The transducers are x-cut quartz disks and resonate at a fundamental frequency of 5 MHz (±1%). The finish is specified by the manufacturer (Valpey Crystal Corporation) as "clear polish" and the diameter of each is 1.000 (+0.000,-0.003) in. The thickness, dictated by frequency, is approximately 0.03 in. Electrodes are evaporated thin films of chrome and gold, complete on one side and over a 5/8 in. concentric circle on the other.

B. DELAY LINE, MARK I

Because this research was developmental in nature, a discussion of the first version of the delay line and its shortcomings is included. The Mark I delay line is pictured in Fig. 1. When in position for filling and during measurements its long axis was oriented vertically.

Although dissolved air has a negligible effect on the speed of sound in a liquid [4], it is desirable to exclude air and so prevent possible bubble formation on the transducers. Before the delay line was filled, the liquid (water) was heated to slightly less than its boiling point to liberate dissolved gases and then allowed to cool to room temperature. The delay line was filled to the top. The top transducer was then inclined slightly to the horizontal and allowed to slip in and sink to its resting surface. It was hoped that this procedure would minimize bubble entrapment on its underside. Excess liquid (above the top transducer) was removed so that the upper surface of the transducer was dry. The top end

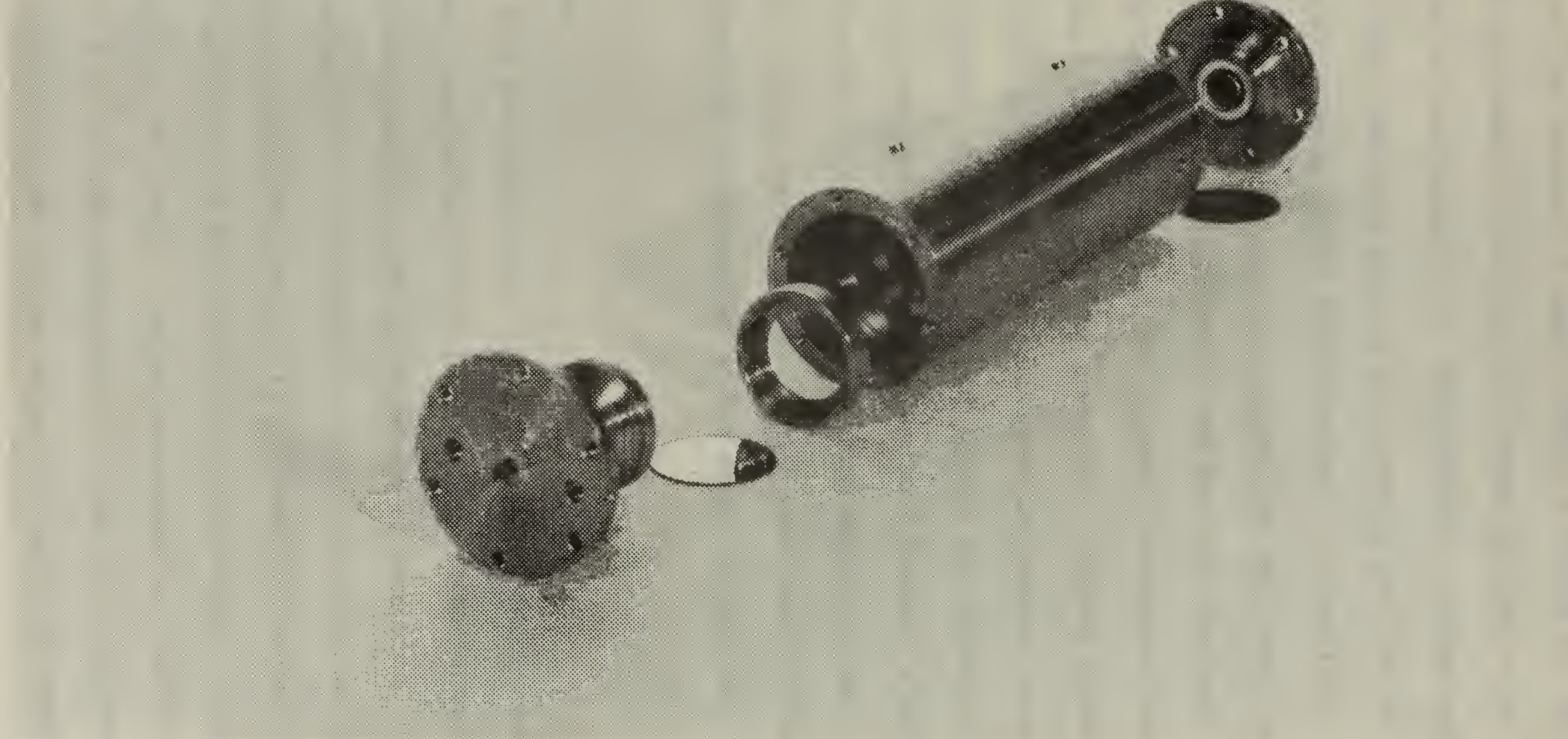


FIGURE 2. DELAY LINE MARK II

of the velocimeter was then sealed. A small amount of liquid was then reintroduced so that the level in the bore would never get below the top transducer during an experimental run. Nevertheless, bubbles would accumulate under the top transducer after a day, causing the loss of a good acoustical contact between liquid and transducer. It was then thought that this problem could be solved by subjecting the liquid to a vacuum to thoroughly degas it, but a second problem then developed which necessitated redesign. Several of the crystal transducers fractured while the velocimeter was completely assembled. Because it was not possible to polish the recessed surfaces bearing on the crystal transducers to a finer tolerance than ordinary machining it was impossible to guarantee that the surfaces were flat to an acceptable degree. Flexural moments resulting from irregularities of the bearing surfaces were probably the cause of the shattered crystals. Both problems of entrapped air and fractured transducers were solved by the design of the Mark II delay line.

C. DELAY LINE, MARK II

Free-machining #303 stainless steel was the material used in both versions of the delay line and was chosen because this chrome-nickel alloy has good machining characteristics and is resistant to corrosion. Figure 2 shows this configuration of the Mark II delay line which is pictured in Fig. 3. The liquid under investigation is contained in the bore which is filled half way to the top of the outer delay line jacket; this allows for volume variations in the liquid. Nominal dimensions of the delay line are included in Fig. 2. The velocimeter is oriented horizontally so that any bubbles which form might collect along the top of the bore and not interfere with a good acoustic bond between liquid and transducer.

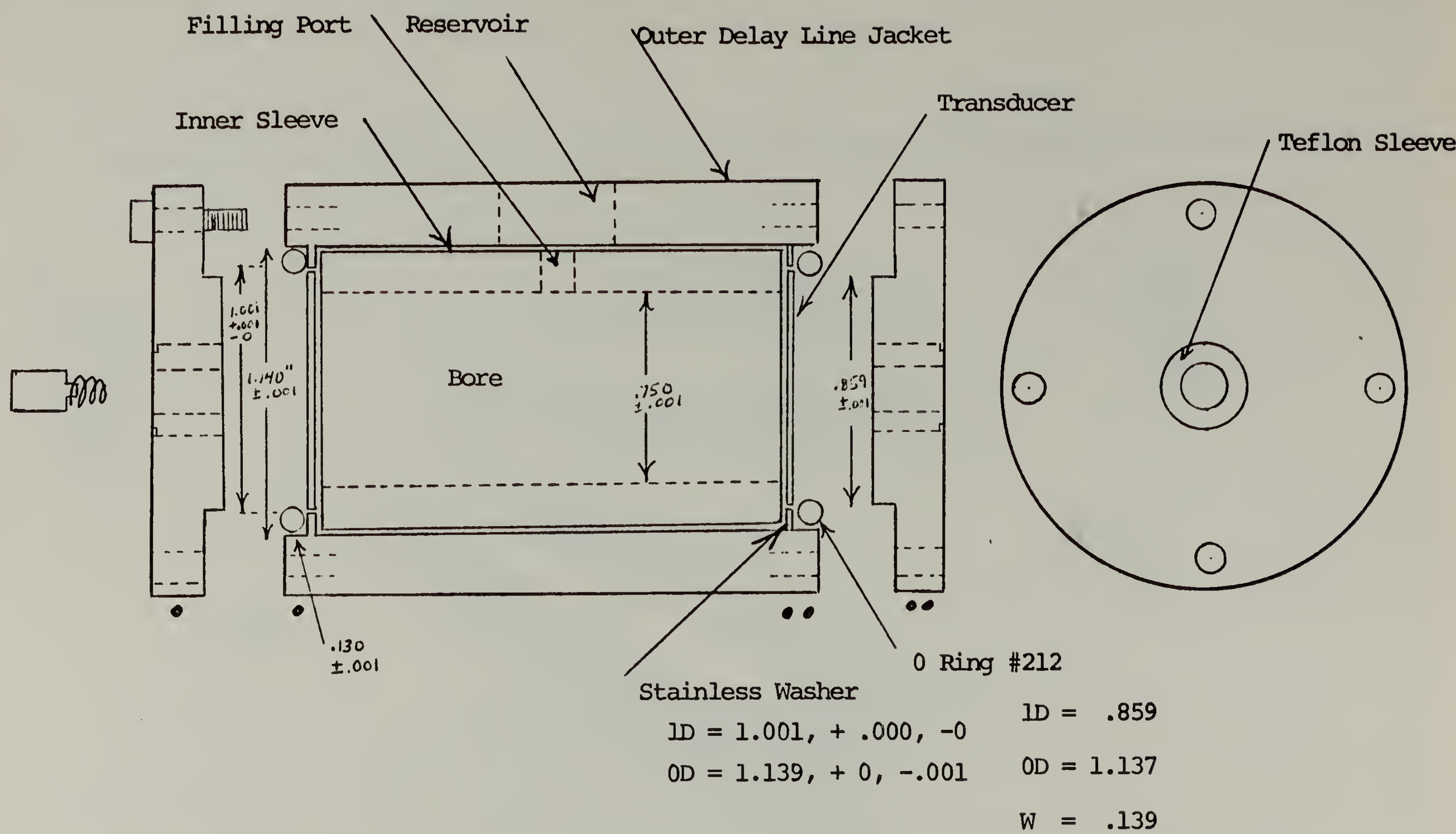


FIGURE 2. MARK II DELAY LINE ASSEMBLY

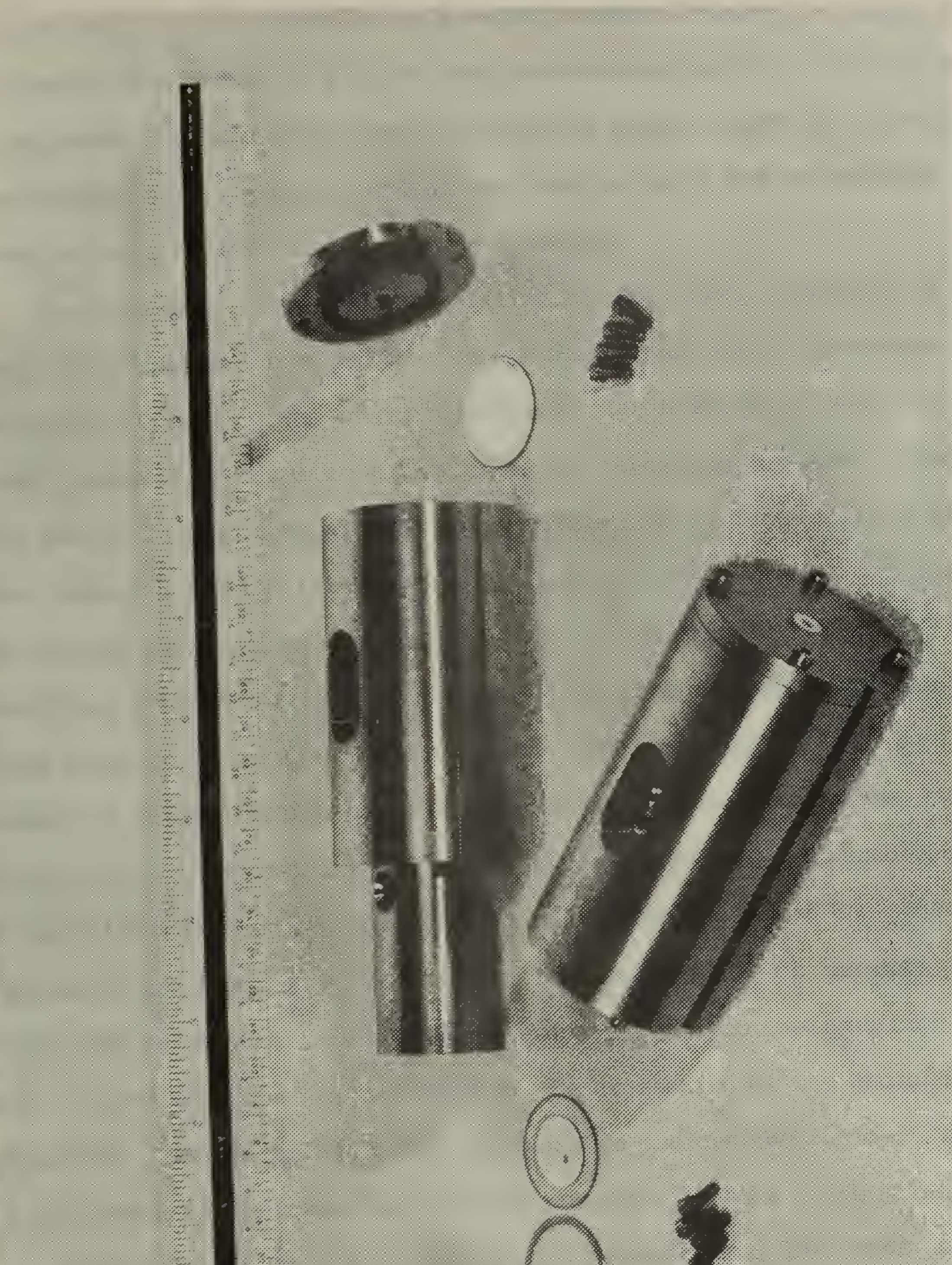


FIGURE 3. DECAY LINE MARK II

Bubbles were not a problem in this second version because the liquid was subjected to a vacuum by placing both loaded velocimeters in a bell jar after which the delay lines were alternately tilted, first by lowering one end and then the other so that any bubbles which had accumulated near the ends of the delay lines could escape.

The ends of the inner sleeves upon which the crystal transducers rest are highly polished to eliminate stress which might cause transducer fracture. Polishing was accomplished on a flat glass plate (with no jig) with a mixture of barnsite and water as the polishing compound. Through the use of a clear optical flat and a sodium light source Newton's Rings were observed and the interference fringes counted. In the worst case 30 fringes were estimated. This leads to an estimate of $.02 \times 10^{-3}$ in. deviation from planarity for this case. The ends of both inner sleeves were shown to be parallel to within less than one minute of arc by the method of autocolimation using a spectrometer, gaussian eyepiece, and optical flats. Each inner sleeve was placed on a fully silvered optical flat and a half-silvered optical flat was placed on top of the sleeve. The departure from parallelism fo the two facing sides of the optical flats manifests itself as a displacement from perfect superposition of the reflections from the two faces of the crosshairs of the gaussian eyepiece. The spectrometer was then used to measure the angle of this displacement of the cross-hairs under the rotation of a plane mirror.

Because of deviations from identity of dimensions, the outer delay line jackets, pressure vessels, and the thermal jacket are all coded as follows: delay line A has ends coded (.) and (..) and the proper end pieces must be matched; the delay line is inserted with the (..) end first into the similarly marked pressure vessel which is in place in the proper

(right-hand) side of the thermal jacket. Corresponding (.˙.) and (::) are used with delay line B and the (::) end is inserted first. The inner sleeves are interchangeable and the pressure vessels may be interchanged to check on uniformity of temperature provided that pressure connections are not made to the pressure vessels.

Reservoir size was determined by two considerations. Since access to the bore was necessary, the filling port which acts as a part of the reservoir had to be of a size allowing for convenient filling and the escape of gas bubbles. The location of the hole was chosen midway along the delay line so that reflections from the hole would interfere minimally with pulse reflections between the two transducers. From tables of density and compressibility of water as a function of temperature and pressure, the maximum volume reduction at 2000 psi is one percent of the volume at one atmosphere. The maximum expansion due to heating from 25°C to 100°C is 4.1% [5]. To allow a safety factor, a total reservoir volume of 10% of the bore was included, most of which is in the outer delay line jacket. Because of the necessity for a sliding fit between inner and outer cylinders of the delay line, the small interface between the cylinders slowly fills with liquid and absorbs part of the liquid in the reservoir.

All stainless steel parts of the delay line assemblies were chemically cleaned (passivated) in a 20% nitric acid solution containing 5% by weight of sodium-dichromate. This process removed any foreign bodies present as a result of machining and polishing which tend to cause superficial rusting and discoloration.

The delay lines are assembled as follows: (refer to Fig. 2). The inner cylinder is gently slipped through the (..) end and placed on the bench with the (..) end up. The stainless steel washer is then placed

in the recess and the transducer inside it with the fully plated side of the transducer toward the bore. A neoprene "0" ring (Parker compound C-557-7) is placed on top of both the transducer and washer. The (..) end plate is then fastened in place using four allen head screws thus providing axial pressure on the transducer through slight compression of the "0" ring. Care must be taken to ensure that the inner access port is centered in the outer cylinder's reservoir. The delay line is then turned over and the (.) end sealed in similar manner except that the lip at the (.) end replaces the washer. The "0" rings serve to provide liquid seals for the contents of the bore and no other pressure is exerted at these interfaces. The teflon sleeves pressed into the end pieces hold the brass electrical leads and prevent contact with the body of the delay line. Contact is made to the outer electrode of the transducer by a light helical spring.

D. PRESSURE SYSTEM

The two pressure vessels shown in Fig. 4 were fabricated from free-machining #303 stainless steel. Electrical connections in this version are made through the pressure vessel wall by cone-compression type electrical connections manufactured by Aminco. Pressure leaks were a constant problem despite the rated capacity of 20,000 psi. The detailed sketch of the manufacturer's specifications for installation shown in Fig. 5 seems to point to two likely sources of this problem. The first is that the angle at the bottom of the cone is not within the tolerance of 60° (±1°). The second is that the distance from the bottom of the cone to the other side (distance a) is not long enough. On the existing pressure vessels this distance is 0.375 in. A possible solution to the problem is the substitution of either neoprene or nylon for soapstone as the sealing material.

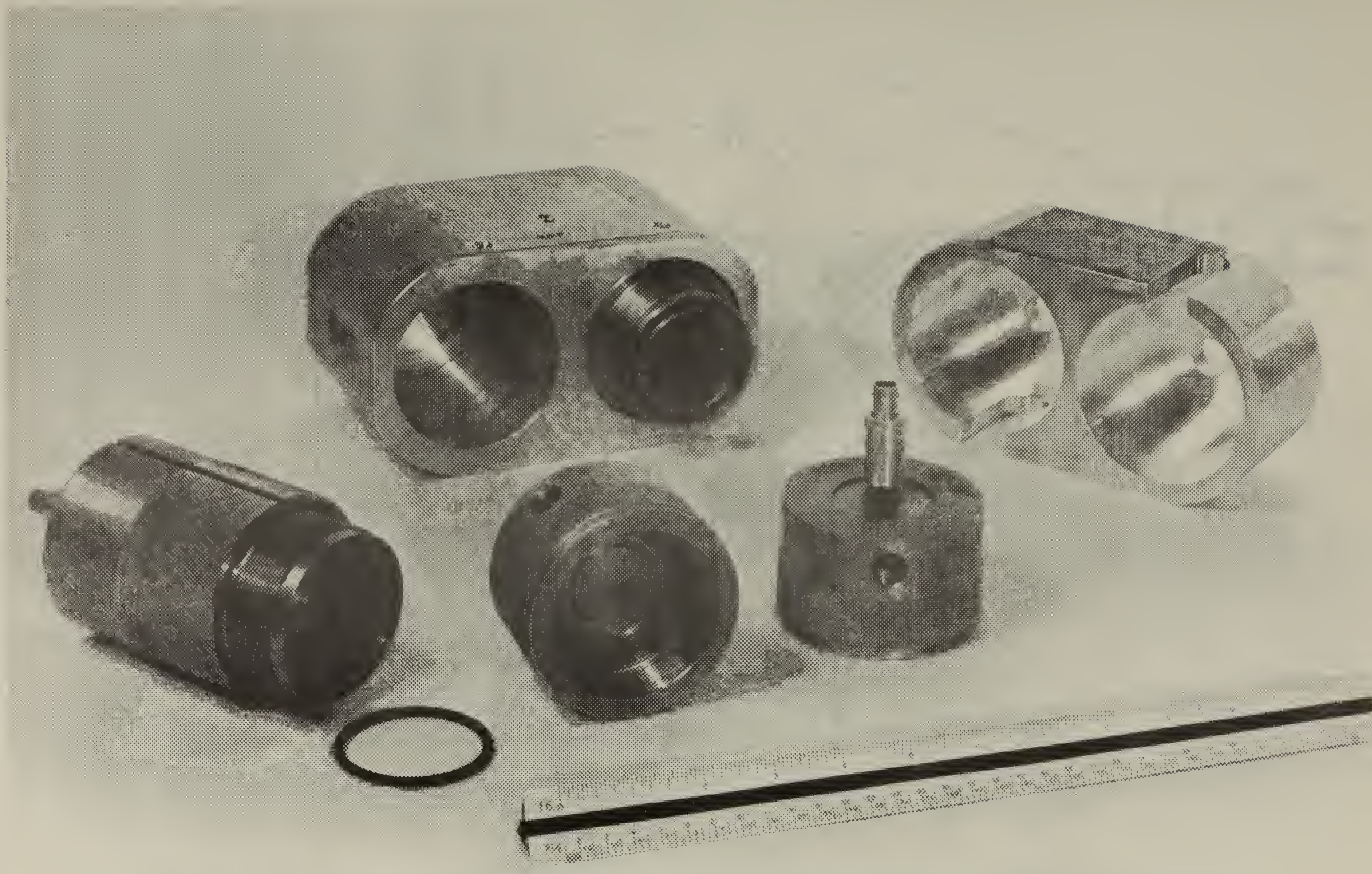


FIGURE 4. PRESSURE VESSELS AND THERMAL JACKET

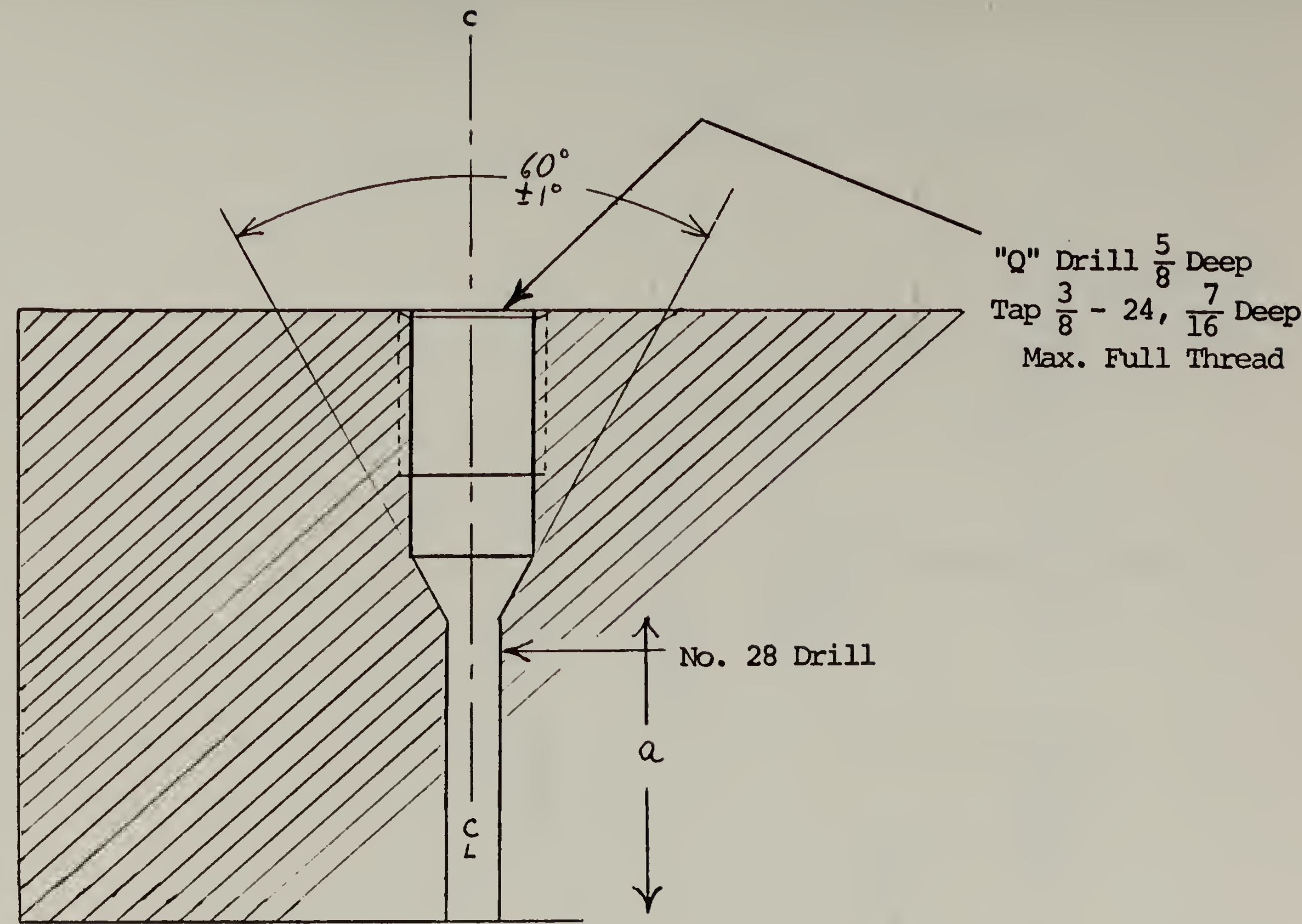


FIGURE 5. SPECIFICATIONS OF CONE COMPRESSION-TYPE ELECTRICAL CONNECTION OPENING

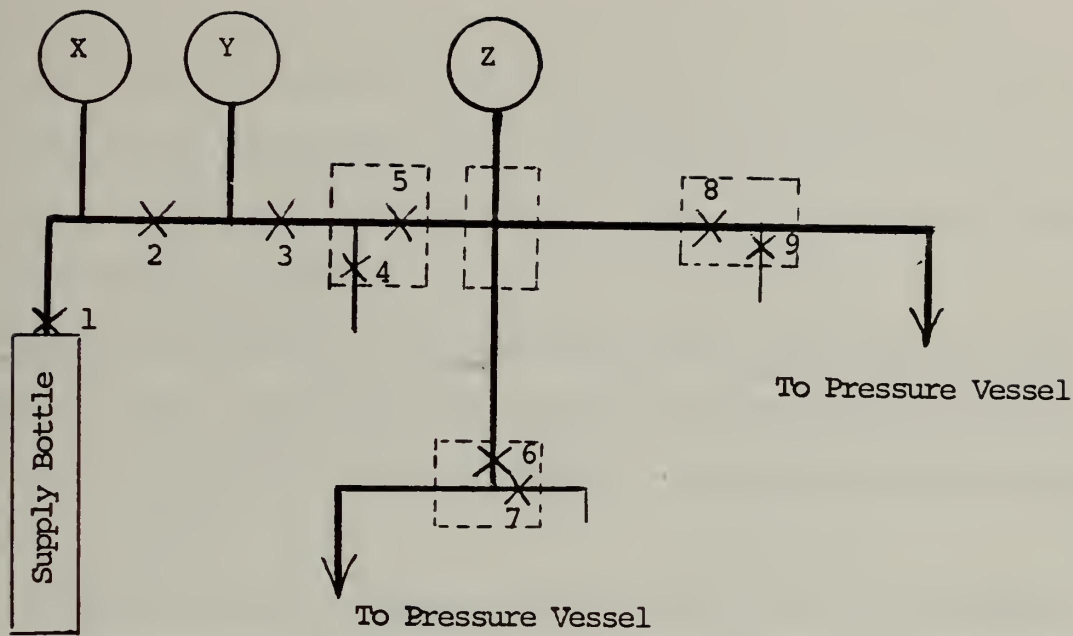


FIGURE 6. PRESSURE SYSTEM SCHEMATIC

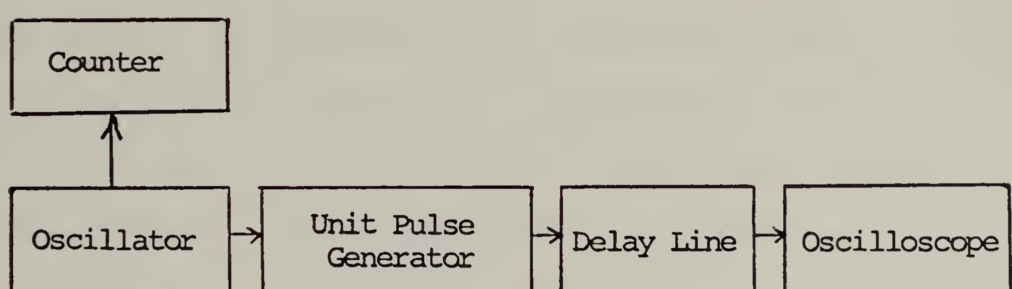


FIGURE 7. ELECTRONICS BLOCK DIAGRAM

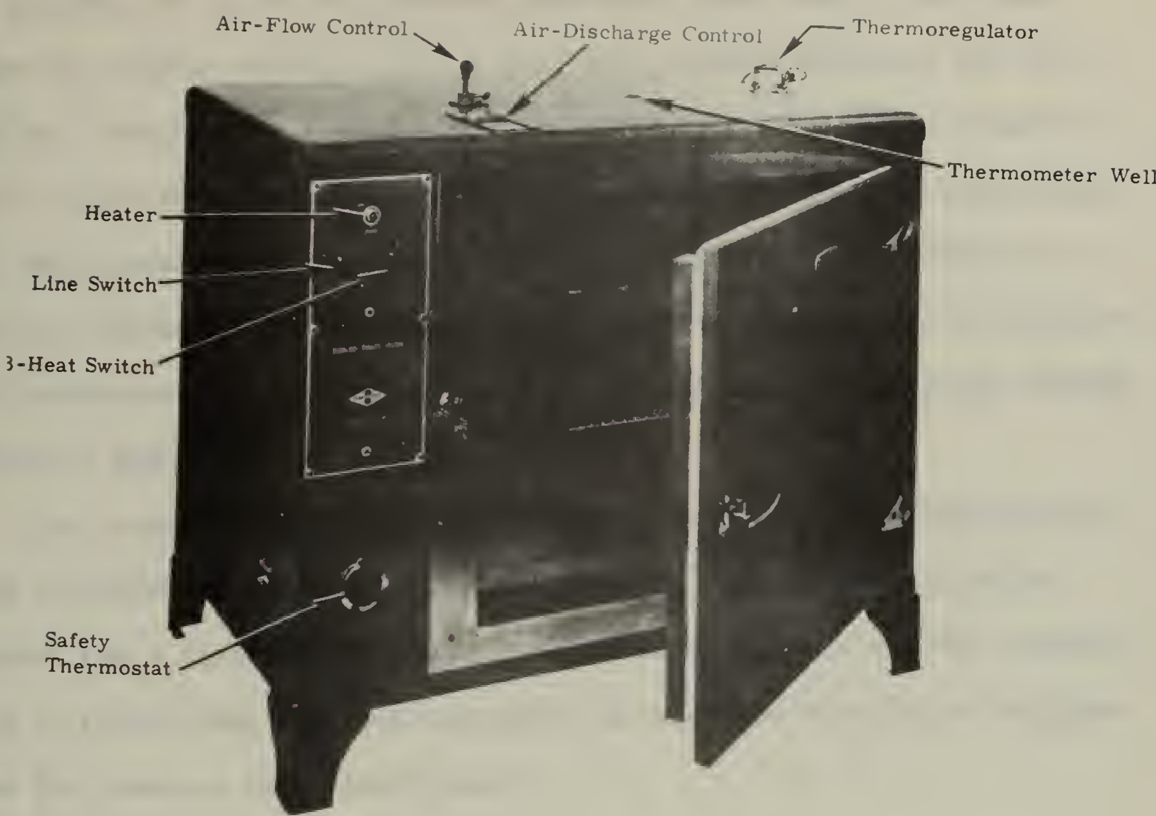
The pressure vessel caps tighten down to a metal-to-metal contact compressing the large "O" rings and no leakage at this interface has ever been noticed. Because of the orientation of the pressure supply line connections inside the oven it is essential that the matching letters (A and B) and matching marks (., .., ∴, ::) be correctly mated on the assembled apparatus.

Pressure is supplied to the pressure vessels from a bottle of compressed nitrogen. The physical connections are made with cone-type and lens ring high pressure tubing connectors. The pressure system schematic is shown in Fig. 6.

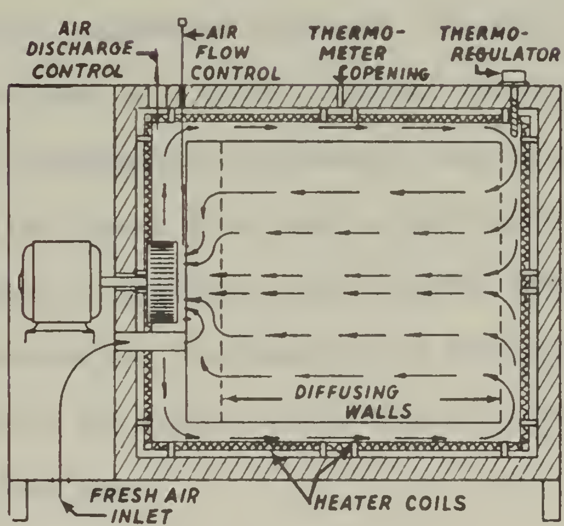
The pressure system is controlled as follows: with both pressure vessels sealed and connected to their supply lines and all valves closed, valve numbers 1, 3, 4, 6, and 8 are opened. Valve number 2 is then slowly opened until the desired pressure is reached throughout the system as indicated on gauges Y and Z. Valve number 4 is then closed and the pressure vessels are isolated from the supply. Gauge X indicated the the amount of pressure left in the supply bottle. Any part of the system may be isolated and have its pressure released without affecting the rest of the system.

E. TEMPERATURE CONTROL SYSTEM

A forced-draft convection oven with a temperature range of 10°C above ambient to 260°C and a stability of ±.5°C provided the heat bath for this research and is shown in Fig. 8. A motor-driven blower produces movement of a large volume of heated air horizontally across the work chamber from right to left after passing it over heaters which circle the inner diffusion walls. This configuration is supposed to distribute uniform heat throughout the entire oven chamber, eliminating hot and cold pockets. As



Aminco Forced-Convection Oven, Cat. No. 4-3540.



Typical Oven Cross-Section, Showing Air Flow

FIGURE 8

is pointed out in the calibration section of this report, the right side is slightly warmer than the left due to this pattern of air flow. The two vessels must be oriented with their long axes in the direction of air flow to ensure that both vessels experience the same temperature. At the suggestion of the manufacturer the damper was kept fully up and the air discharge control slide fully closed to maximize air circulation and thereby assure the greatest degree of temperature uniformity through the work space.

The existence of the thermal gradient inside the oven necessitated the inclusion of the thermal jacket shown in Fig. 4. This aluminum jacket has a sliding fit with the two pressure vessels and the aluminum cap is placed over the open end after the pressure vessels are in place and the pressure connections made.

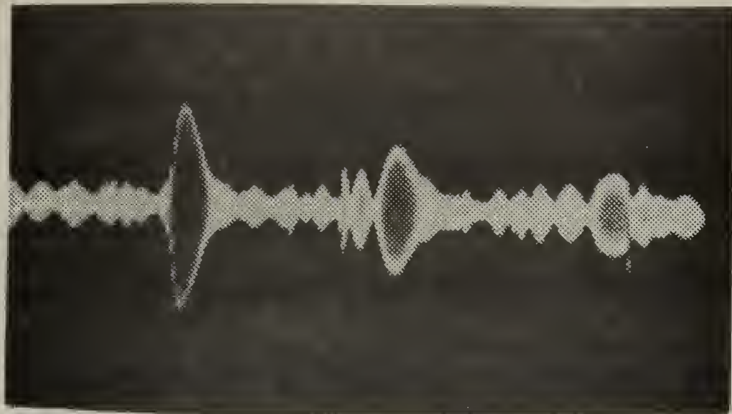
F. TEMPERATURE MEASUREMENT

Temperature measurements were made using a thermistor thermometer using two different, supposedly identical, ten foot vinyl-covered wire probes, each terminated in a three conductor phone plug. Although the two probes had a characteristic difference that is almost linear with temperature, the two probes were used to determine the ΔT's between the two pressure vessels in different oven internal configurations. These calibration procedures are discussed in the calibration section. Two holes are drilled to the middle of the thermal jacket to hold these probes during a data run.

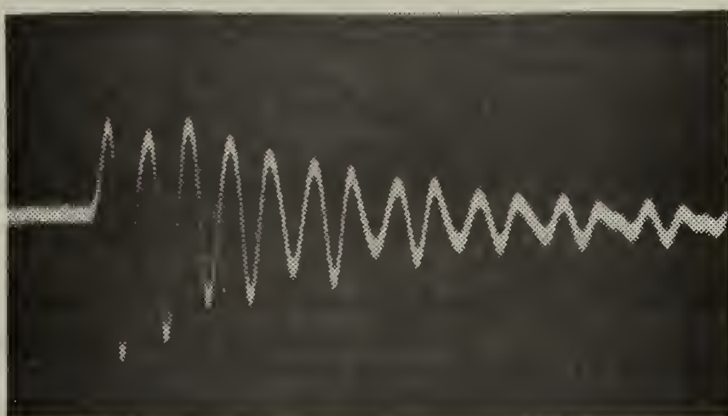
G. ELECTRONICS

The electronics are shown in the block diagram of Fig. 7. The sine wave oscillator was used as an external driving source for the unit pulse generator. Rise time of the unit pulse triggered by each positive half cycle of the sine wave was typically 12 nanoseconds. The pulse used was typically about 0.1 μ sec in duration but this duration was lengthened to minimize all but the first two half cycles of the superimposed echoes as displayed on the CRO.

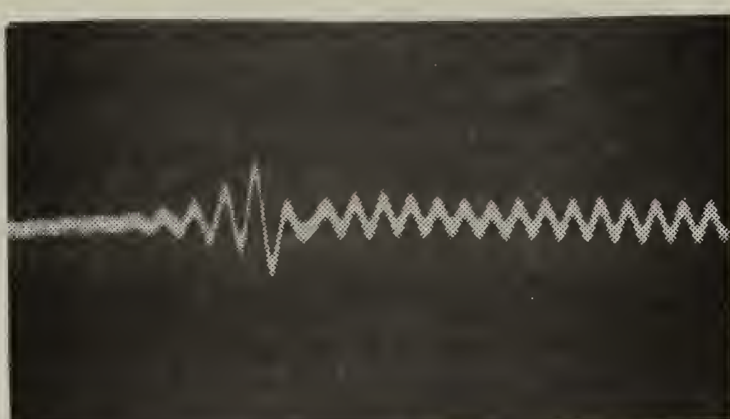
The receiving transducer generates the electric signal which is then displayed on the CRO. The sweep of the oscilloscope is externally triggered by a negative pulse from the unit pulse generator. The delayed time base capability of the oscilloscope makes possible a convenient display that simplifies superposition of all echoes. The wave forms are shown in Fig. 9. Coincidence is achieved as follows: at a moderate sweep and at a frequency slightly greater than that for coincidence the pulse and successive echoes are displayed. The frequency is then lowered with the fine tuning adjustment until the peak of either the first or second half cycle is maximized. Care must be exercised so that the frequency is not lowered below the frequency of superposition or the echoes will "leak through". The frequency of superposition which is related to the speed of sound as noted in the introduction may then be read directly from the electronic counter. The technique of superposition requires much practice, utilizing expanded horizontal and vertical displays of the oscilloscope presentation and very careful adjustments of the fine tuning adjustment on the driving oscillator.



FIRST THREE REFLECTIONS AT REPETITION
RATE GREATER THAN THAT FOR SUPERPOSITION



COINCIDENCE OF ALL ECHOES BY
MAXIMIZATION OF FIRST HALF CYCLE



FREQUENCY LOWER THAN THAT FOR
SUPERPOSITION AS EVIDENCED BY "LEAK THROUGH"

FIGURE 9

III. CALIBRATION

A. COMPARISON OF THERMISTOR PROBES

Of the two probes used in conjunction with the digital thermometer, probe B always read higher and the difference between the two probes is seen to be a nearly-linear function of temperature (Fig. 10). It did appear that there was a time-dependent change in the function as well. The purpose of determining this difference between probes at different temperatures was to enable the thermometer to be used to compare temperature condition of the two velocimeters. During these calibrations runs both probes were led into one pressure vessel through a two-hole rubber stopper.

B. ORIENTATION OF PRESSURE VESSELS IN OVEN

As indicated in the discussion of the forced-draft oven, temperature gradients are present inside the oven. With one probe in each pressure vessel, temperature measurements were made of the interior temperatures of the pressure vessels. Based on preliminary data from the probe calibration discussed above, it was concluded that a thermal jacket surrounding both pressure vessels would be required to minimize any temperature gradient between the two velocimeters. The problem then became one of orientation of the thermal jacket holding the pressure vessels.

It was then discovered that the tabulated temperature difference between probes was dependent upon whether one or both of the probes were in contact with the walls of the pressure vessels. Because of this contact problem two small aluminum blocks that would fit inside the pressure vessels were fabricated. Each block had a hole in it that offered a

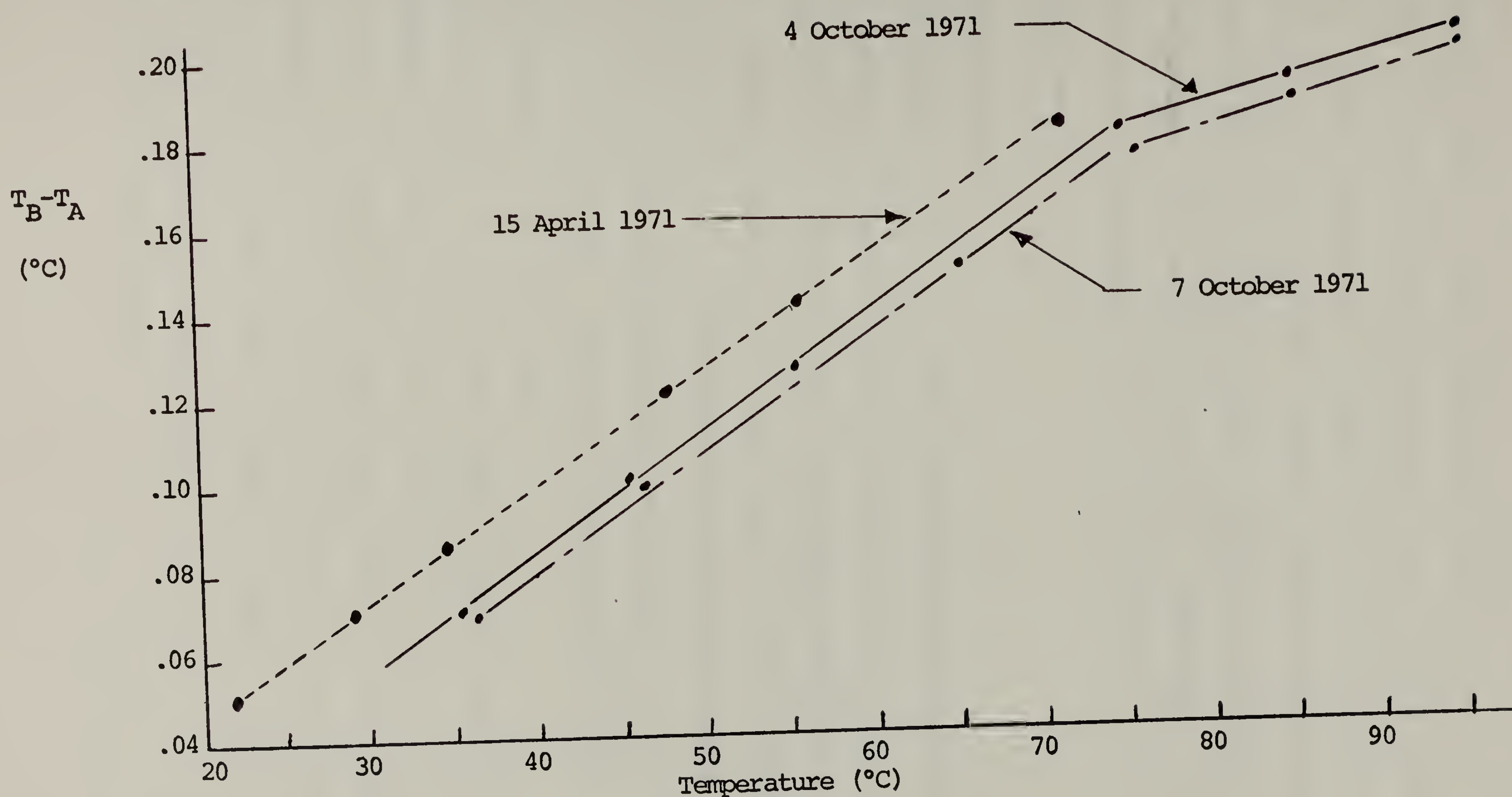


FIGURE 10. PROBE CALIBRATION CURVES

sliding fit to the thermistor probe tip. The probes were led into the pressure vessels through a rubber stopper and into the holes in the blocks. Then the system was cycled from 35°C to 95°C in ten degree increments. At each temperature the probes were exchanged and the system was allowed to come to equilibrium. Several orientations were examined until one was found for which T_B-T_A before exchange equaled T_B-T_A after exchange within the least reading of the digital thermometer, 0.01°C. This meant that any temperature gradient that existed had to be on the order of, or less than, 0.005°C.

C. SYSTEM CALIBRATION

To demonstrate that the velocimeter system does precisely and consistently measure the speed of sound in a liquid, two data runs were made. Each consisted of two complete cycles in ten degree increments from 35°C to 85°C. Distilled water was the liquid chosen for these measurements and on each run the water on both sides was from the same sample.

On the first run the pressure vessels were placed in their proper sides of the thermal jacket. The system was cycled once, allowed to cool to room temperature, and cycled again.

Before the second run the pressure vessels were disassembled, cleaned, and reassembled. A different sample of distilled water was then used to fill both sides. The pressure vessels were placed in their proper sides of the thermal jacket and the system was cycled once and allowed to cool to room temperature. Then, without opening the pressure vessels, they were exchanged and the system was cycled again. Data from these two runs are presented in the results section.

IV. RESULTS

The following is a tabulation of average values of frequencies of superposition with calculated rms error. The ratios between the two frequencies for the two velocimeters are also presented. The indicated temperatures are the nominal values at which the data were obtained.

RUN I, CYCLE 1

TEMP	ν_A	ν_B	ν_A/ν_B
5	10.50009±27	10.49973±28	1.000034±38
45	10.60229 28	10.60199 26	1.000028 37
55	10.66839 41	10.66822 24	1.000015 45
65	10.70868 30	10.70833 23	1.000042 35
75	10.71552 48	10.71515 39	1.000034 58
85	10.69337 44	10.69327 33	1.000009 51

RUN I, CYCLE 2

TEMP	ν_A	ν_B	ν_A/ν_B
35	10.47999±41	10.47944 22	1.000052 44
45	10.59117 38	10.59088 34	1.000027 48
55	10.66815 34	10.66783 38	1.000029 48
65	10.70982 43	10.70949 23	1.000030 45
75	10.71659 24	10.71626 11	1.000030 25
85	10.69338 15	10.69325 19	1.000012 34

RUN II, CYCLE 1 (BEFORE EXCHANGE)

TEMP	ν_A	ν_B	ν_A/ν_B
35	10.47815±37	10.47771 30	1.000041 45
45	10.59757 16	10.59734 20	1.000021 24
55	10.67233 21	10.67205 17	1.000026 26
65	10.70851 22	10.70802 22	1.000045 29
75	10.71729 29	10.71699 29	1.000027 38
85	10.69821 30	10.69752 26	1.000064 37

RUN II, CYCLE 2 (AFTER EXCHANGE)

TEMP	ν_A	ν_B	ν_A/ν_B
35	10.49442±32	10.49406 19	1.000034 36
45	10.59747 29	10.59717 21	1.000028 34
55	10.67092 38	10.67045 34	1.000044 48
65	10.70157 17	10.70114 15	1.000040 21
75	10.71594 35	10.71563 27	1.000029 41
85	10.69517 27	10.69444 10	1.000068 27

All data were included in the calculations of rms errors, even though some were outside two standard deviations, because of the small number of data. Consequently, the deviations shown are somewhat larger than those that would be calculated if those apparently spurious ones were left out in accordance with standard statistical procedure for large samples.

V. DATA EVALUATION AND CONCLUSIONS

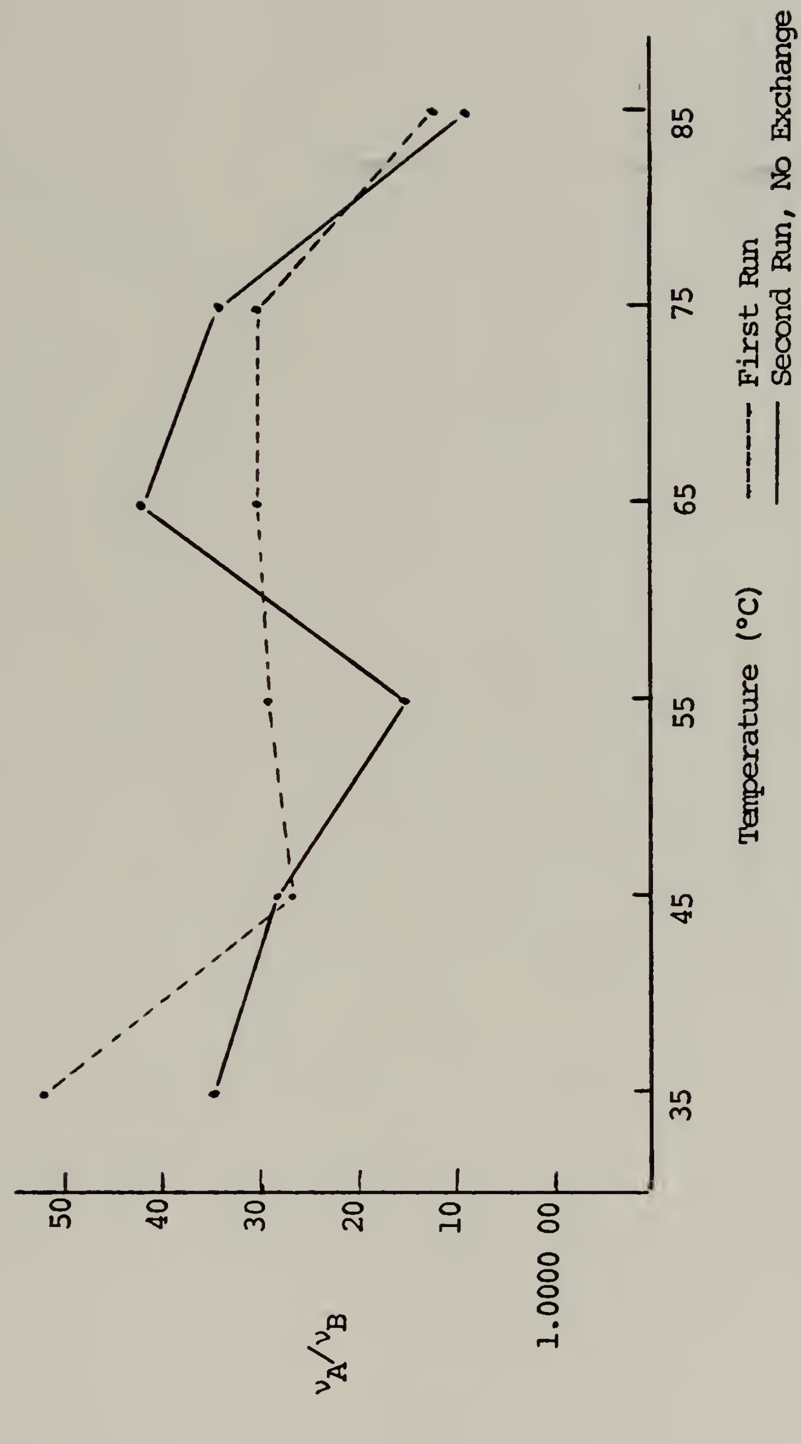
Between 35°C and 85°C we can see from Fig. 11a that the maximum deviation in ν_A/ν_B appeared to be less than 2×10^{-5} in the worst case and 0.9×10^{-5} on the average for the two velocimeters in Run I for which there was no exchange of position. The calculated average rms error in superposition frequencies for each velocimeter is ±0.0003 Hz which corresponds to a calculated uncertainty in the ratio ν_A/ν_B of about 4.5×10^{-4} greatly in excess of the observed uncertainty. The explanation for this discrepancy is not known.

Figure 11b shows the effect of the exchange of the pressure vessels within the thermal jacket on the ratio ν_A/ν_B. The data indicate this ratio is independent of configuration inside the thermal jacket, at least within a statistical uncertainty of less than 2×10^{-5}. This suggests that no observable thermal gradient (within our measurement capability) exists between the two sides of the thermal jacket.

From Fig. 11c it is observed that between 35°C and 75°C the deviation from the average of Fig. 11a by the ratio ν_A/ν_B for a different water sample in the same configuration is easily less than 0.9×10^{-5}. This ignores the data at 85°C which show an unusually large disagreement. Even so, this discrepancy is about 5×10^{-5} which is approximately the same as the calculated average rms error discussed in the first paragraph of this section. The system may therefore be disassembled and reassembled and consistent results obtained.

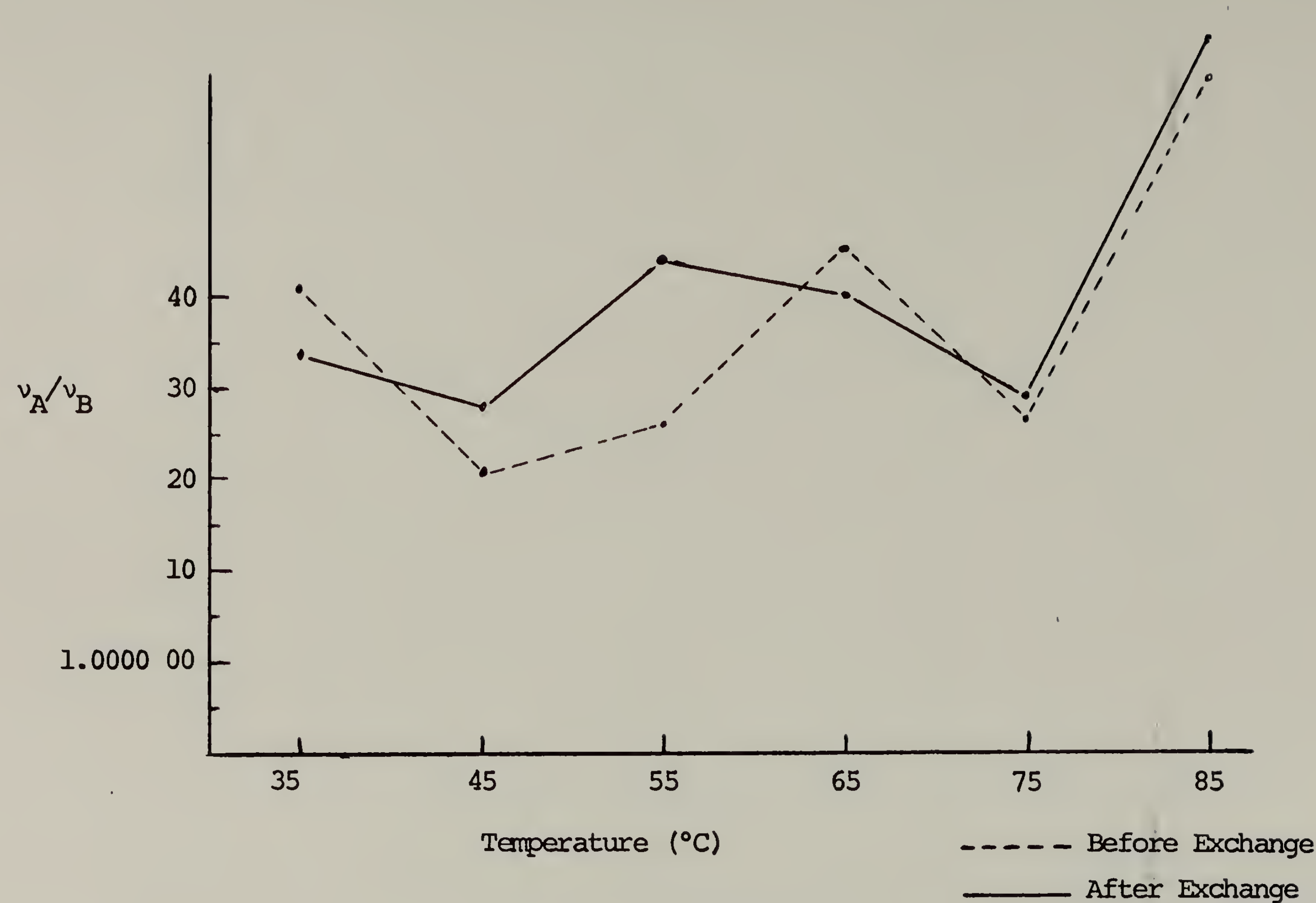
Uncertainty in the difference between two speeds of sound (for two liquids) resulting from the uncertainty in the ratio of lengths of the two velocimeters can be approximated by the observed or calculated fluctuation in ν_A/ν_B multiplied by the nominal speed of sound [6]. This corresponds to an observed uncertainty of $(0.9 \times 10^{-5}) \times (1.5 \times 10^5) =$ 1.3 cm/sec and a calculated uncertainty of about 7 cm/sec. The error in the speed of sound resulting from a temperature gradient of 0.005°C would amount to about 1 cm/sec for water at about 40°C. This upper limit in uncertainty is smaller than the other uncertainties.

The precision of the electronic counter is seen to be sufficient for the measurements of this research. A requirement for more precise control of the driving oscillator is apparent because a very small change of the fine tuning control typically causes a Δf of 0.0010 KHz. This situation could be improved with the addition of a third vernier control for fine tuning.



ν_A/ν_B
1.0000 00
10
20
30
40
50
35
45
55
65
75
85
Temperature (°C)
First Run
Second Run, No Exchange

FIGURE 11a



40
30
20
10
1.0000 00
ν_A/ν_B
35
45
55
65
75
85
Temperature (°C)
Before Exchange
After Exchange

FIGURE 11b

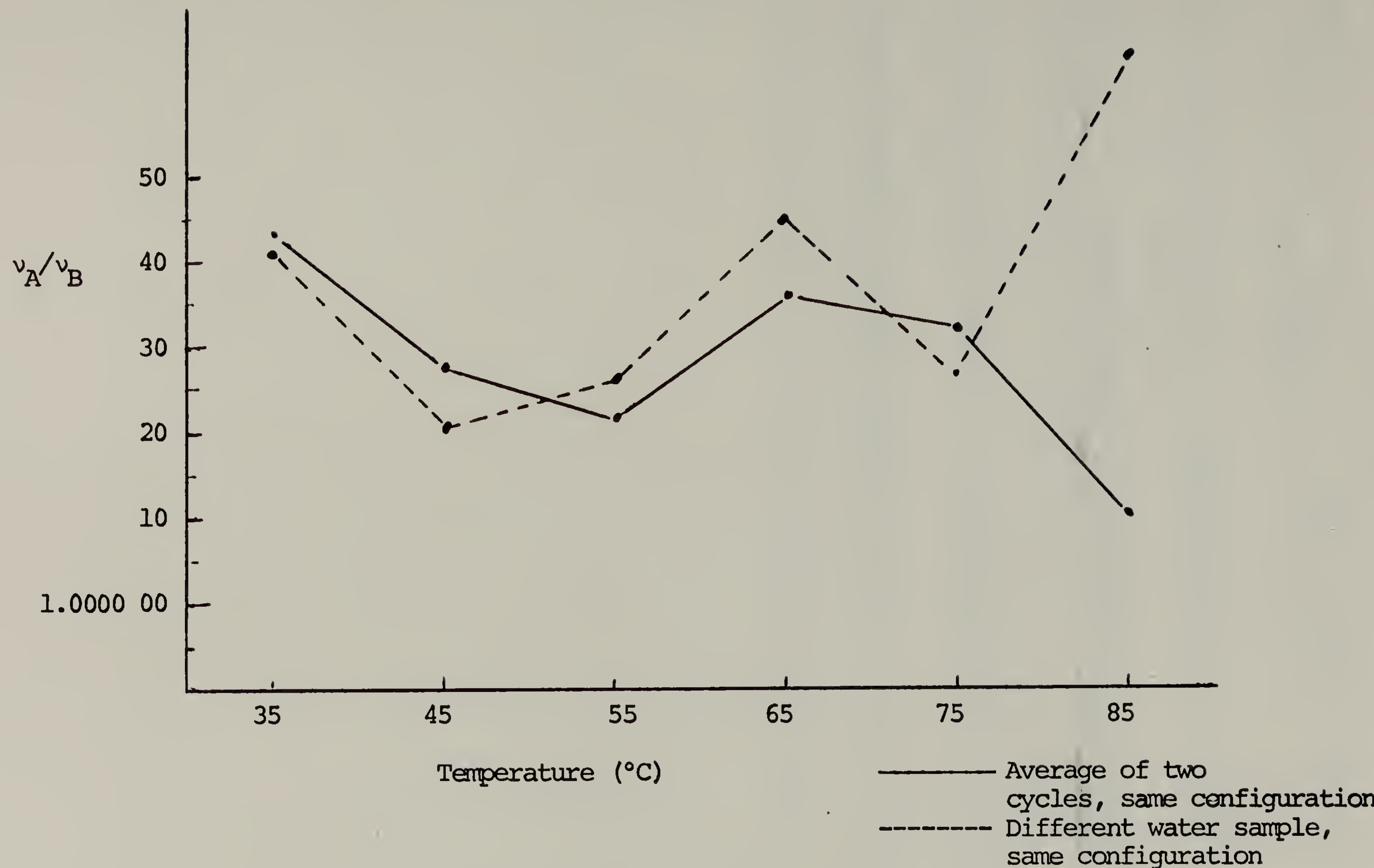

50
40
30
20
10
1.0000 00
ν_A/ν_B
35
45
55
65
75
85
Temperature (°C)
Average of two cycles, same configuration
Different water sample, same configuration

FIGURE 11C

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ABSTRACT

An ultrasonic velocimeter system utilizing two nearly identical velocimeters was developed that could measure the differences in speeds of sound between pure liquids and solutions over the pressure range ambient to 2000 psi and the temperature range ambient to 100°C. Accuracy of two parts in 10^{-6} in frequency corresponding to an average deviation in the speed fo sound of .3 cm/sec is demonstrated.

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